

SCA Wettability Short Course



Advanced measurements of wettability: Pore Scale Imaging



Dr. Matthew Andrew
Oil and Gas Technologist
Carl Zeiss Microscopy GmbH

- 1 Introduction – Why look at the pore scale?**
- 2 Measuring spatially resolved contact angles**
- 3 One application in depth – a heterogeneous carbonate**
- 4 New developments – towards automation**
- 5 Applications – flow, real systems and curvature**
- 6 Other technologies – cryo SEM**

Who am I?



Undergraduate and Masters from Queens' College, Cambridge in Geological Sciences. PhD from Imperial College in Petroleum Engineering, where I developed the first reservoir condition in situ rig, integrated with the Versa XRM, allowing for pore-scale imaging of multiphase flow.

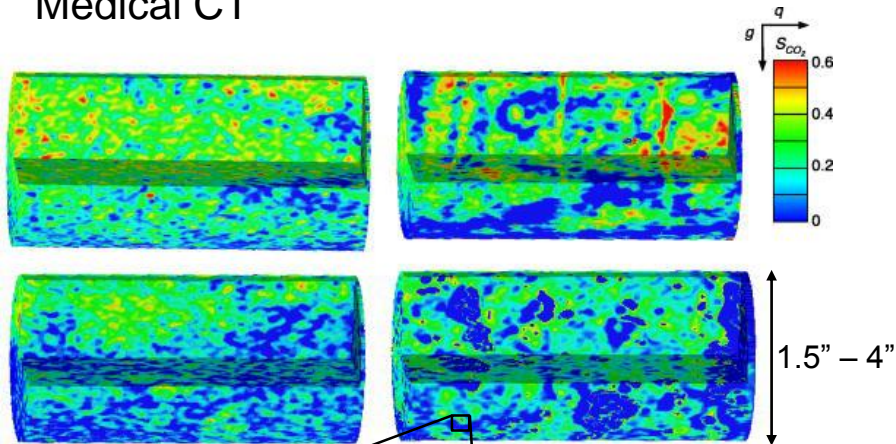
Now I direct O&G & Geoscience within Zeiss Microscopy

- Menke, Reynolds, Andrew et al., 2017. 4D multi-scale imaging of reactive flow in carbonates: Assessing the impact of heterogeneity on dissolution regimes using streamlines at multiple length scales. *Chemical Geology* (In Press)
- Andrew et al. 2018. The Usage of Modern Data Science in Segmentation and Classification: Machine Learning and Microscopy
- Andrew, M., Bijeljic, B. & Blunt, M.J., 2013. Pore-scale imaging of geological carbon dioxide storage under in situ conditions. *Geophysical Research Letters*, 40(15), pp.3915–3918.
- Andrew, M., Bijeljic, B. & Blunt, M.J., 2014. New frontiers in experimental geoscience : X-ray microcomputed tomography and fluid flow., *Microscopy and Analysis* (February), pp.4–7.
- Andrew, M., Bijeljic, B. & Blunt, M.J., 2014a. Pore-by-pore capillary pressure measurements using X-ray microtomography at reservoir conditions: Curvature, snap-off, and remobilization of residual CO₂. *Water Resources Research*, 50, pp.8760–8774.
- Andrew, M., Bijeljic, B. & Blunt, M.J., 2014b. Pore-scale contact angle measurements at reservoir conditions using X-ray microtomography. *Advances in Water Resources*, 68, pp.24–31. Available at: <http://dx.doi.org/10.1016/j.advwatres.2014.02.014>.
- Andrew, M., Bijeljic, B. & Blunt, M.J., 2014c. Pore-scale imaging of trapped supercritical carbon dioxide in sandstones and carbonates. *International Journal of Greenhouse Gas Control*, 22, pp.1–14. Available at: <http://dx.doi.org/10.1016/j.ijggc.2013.12.018>.
- Andrew, M.G. et al., 2015. The Imaging of Dynamic Multiphase Fluid Flow Using Synchrotron-Based X-ray Microtomography at Reservoir Conditions. *Transport in Porous Media*, 110, pp.1–24.
- Menke, H.P. et al., 2015. Dynamic Three-Dimensional Pore-Scale Imaging of Reaction in a Carbonate at Reservoir Conditions. *Environmental Science & Technology*, p.150323102837000. Available at: <http://pubs.acs.org/doi/abs/10.1021/es505789f>.

Continuum vs. Pore Scale



Continuum Scale Medical CT

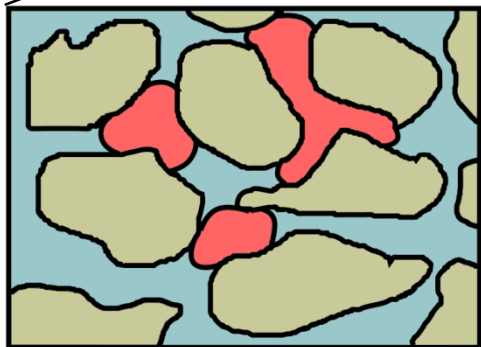


Krevor *et al.* 2012

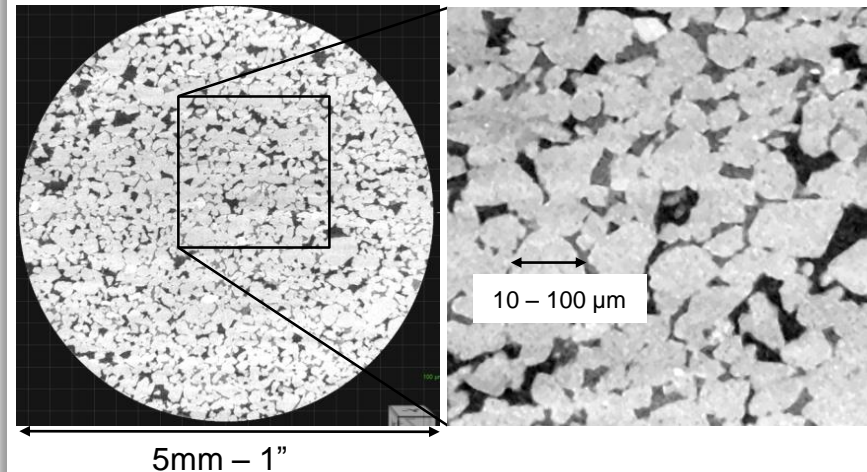
Voxel Size: 0.5mm

Each voxel consists of averaged property, giving a saturation distribution.

This saturation can then be averaged across the core, to form a relative permeability curve, or can be associated with core scale features affecting flow (e.g. sedimentary structures / bedding layers)



Pore Scale XRM

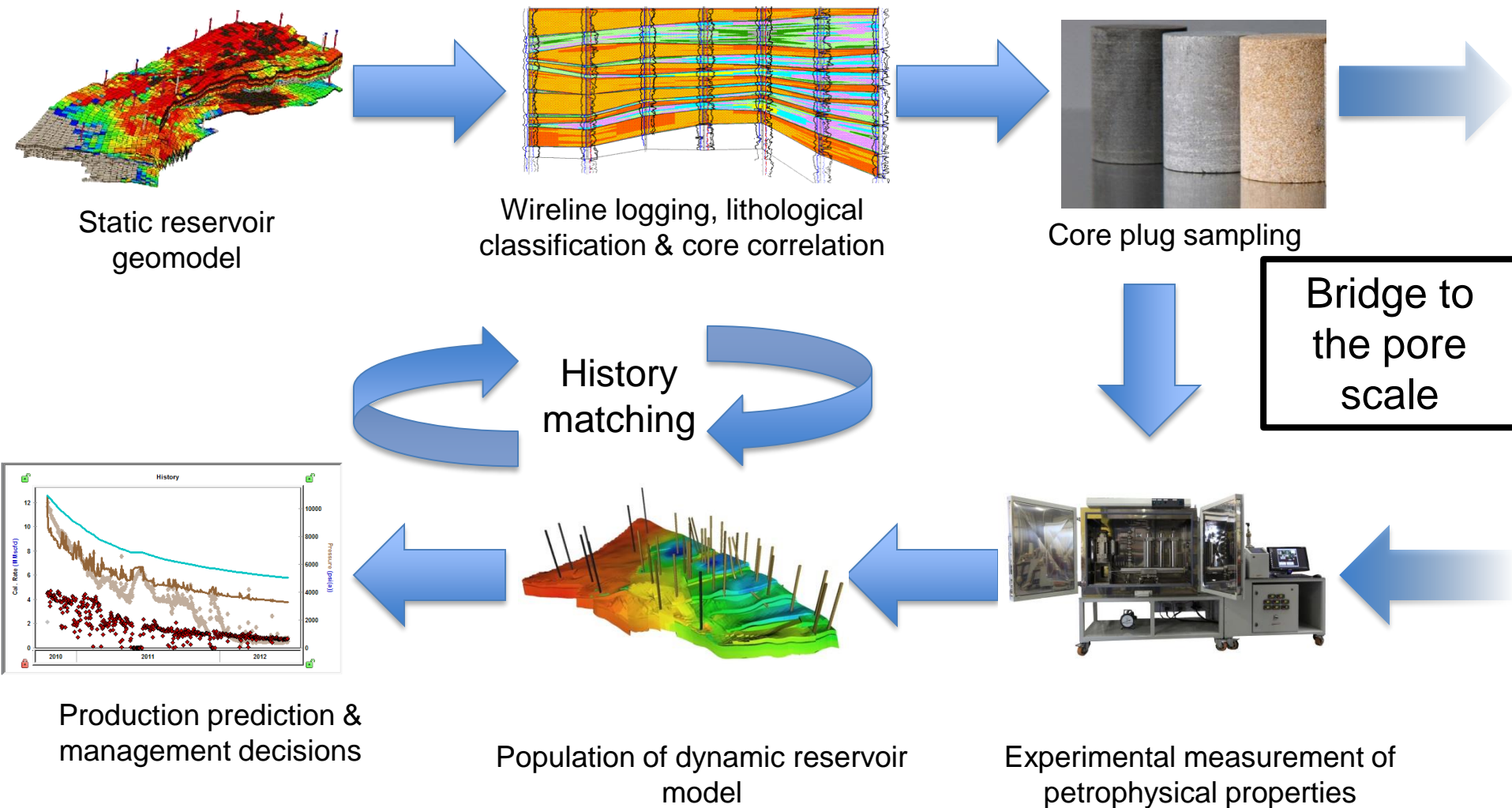


Voxel Size: <1μm

Each voxel uniquely describes a single phase (e.g. rock grain, oil or brine), giving truly pore-scale information.

This information can be used to examine connected vs. disconnected saturation, wettability changes, changes in saturation structure or a wide range of different phenomena fundamentally controlling macroscopic flow and transport phenomena.

Extensive Existing Paradigm for Dealing with Reservoir Scale Heterogeneity



Pore scale vs. grain scale: Fluid flow rooted at the pore scale

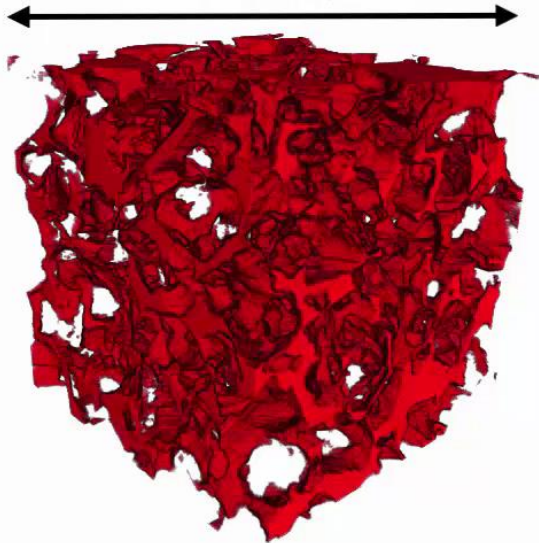


Flow fundamentally rooted at the scale of the pore throat

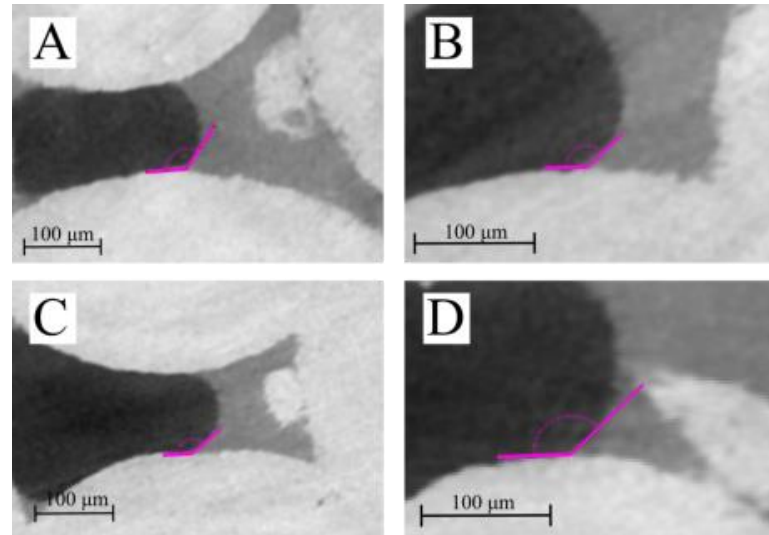
Single phase: Hagen–Poiseuille equation

Porous Medium Scale

100s to 1000s of μm



Multi-phase: Governs pore snap-off & invasion capillary pressure



Threshold capillary pressures:

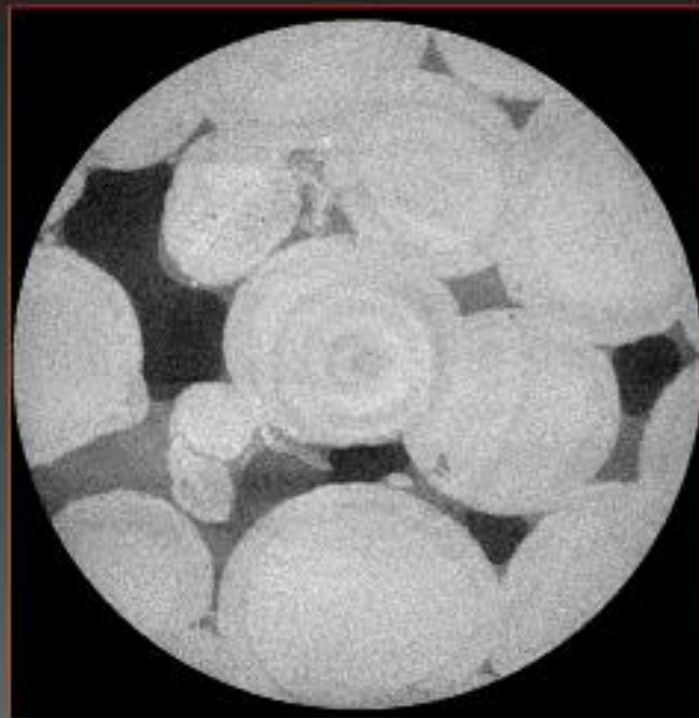
Drainage:
$$P_{cow} = \frac{2\sigma_{ow}\cos\theta_{owr}}{r},$$

Imbibition:
$$P_{c-so} = \frac{1}{r} \times \sigma \left(\cos\theta_a - \frac{2\sin\theta_a}{\cot\beta_1 + \cot\beta_2} \right)$$

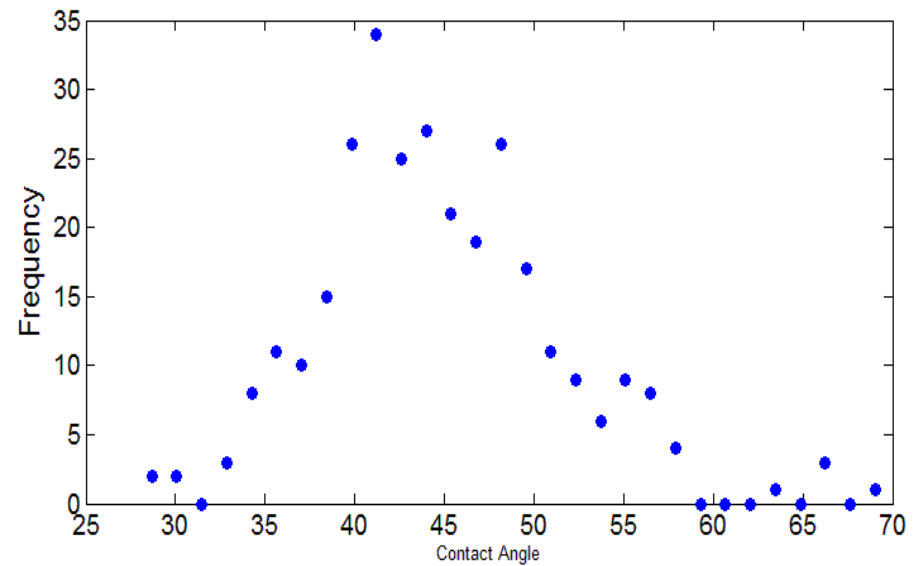
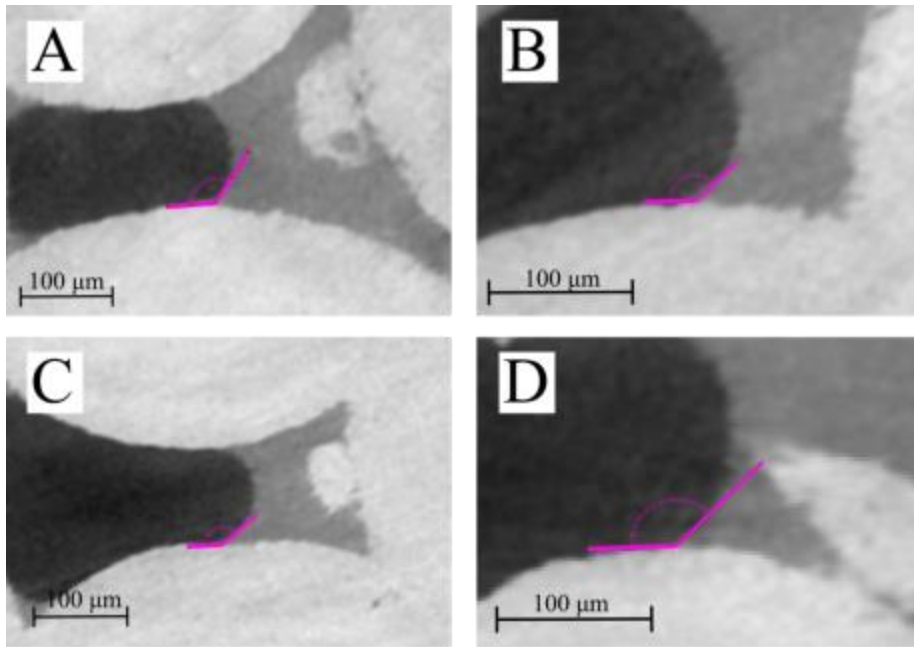
Experiments



Contact angle measurement



Measuring on real systems



$45 \pm 10^\circ$

Contact angle measured directly on the resampled dataset.

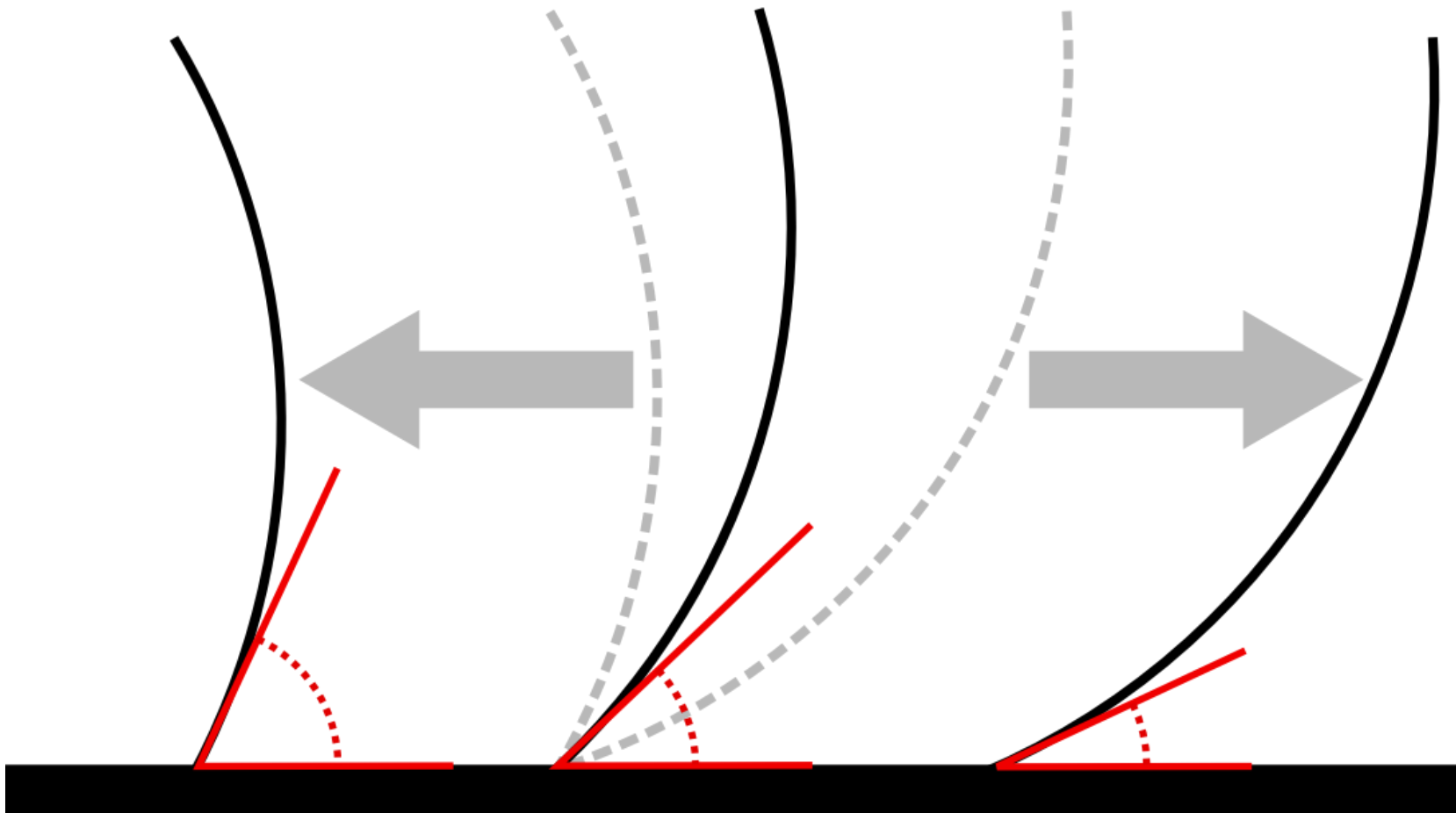
Contact angle distribution – contributing factors



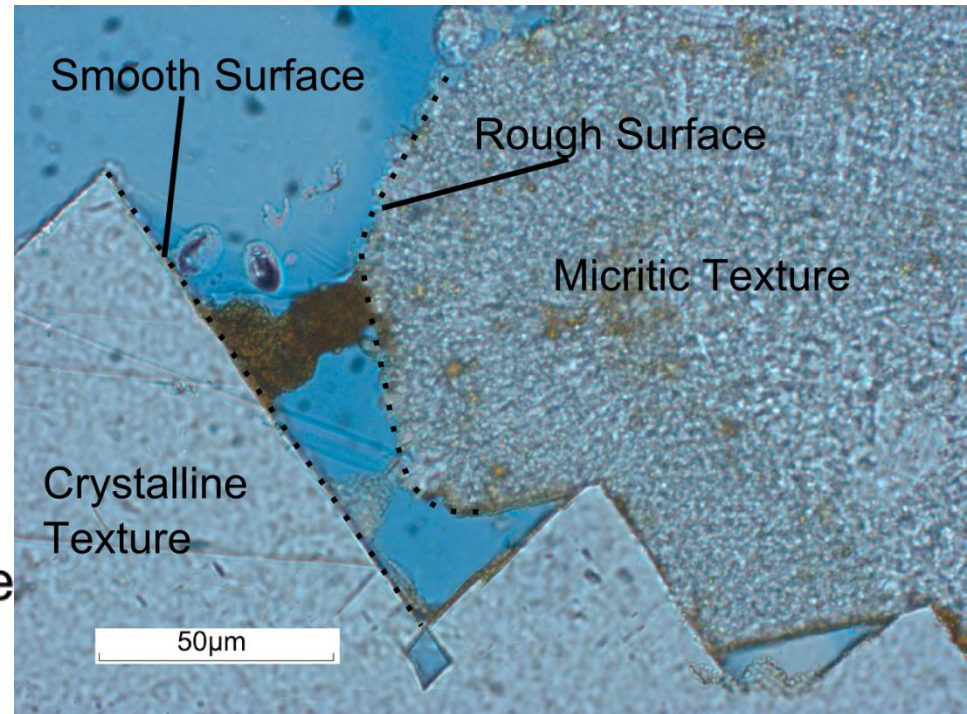
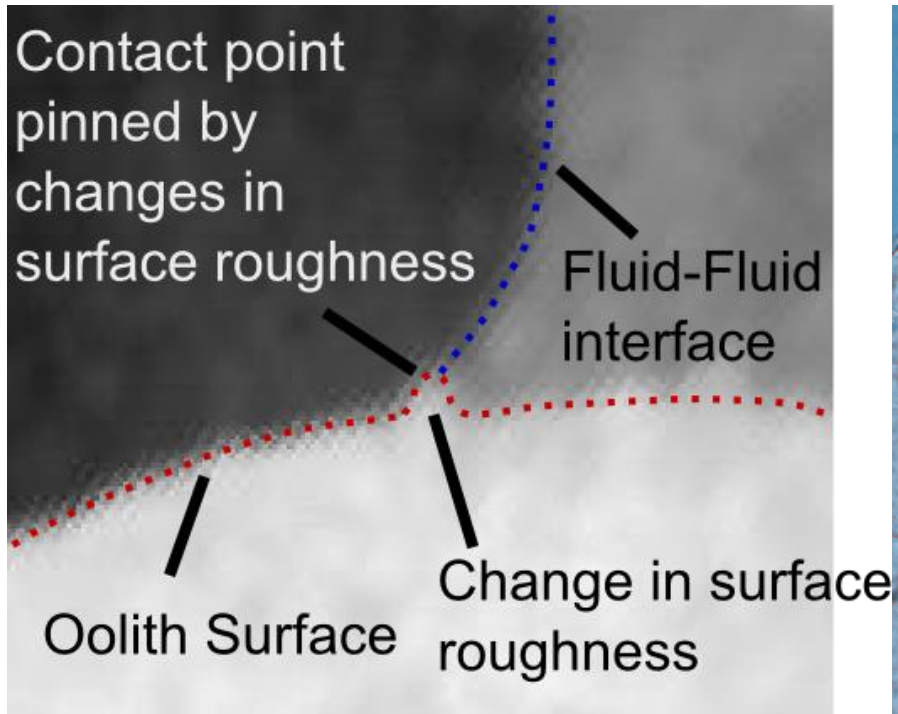
Advancing

Equilibrium

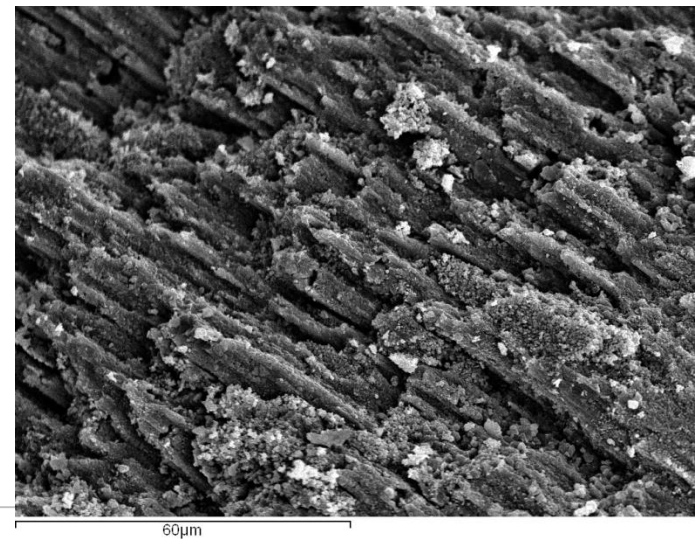
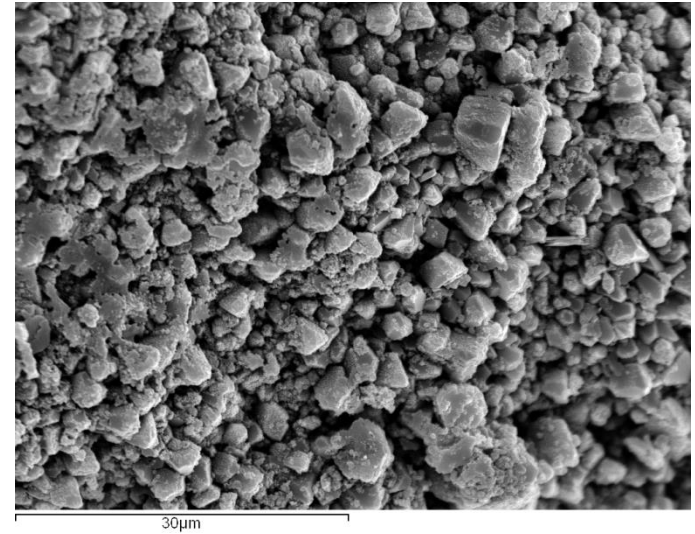
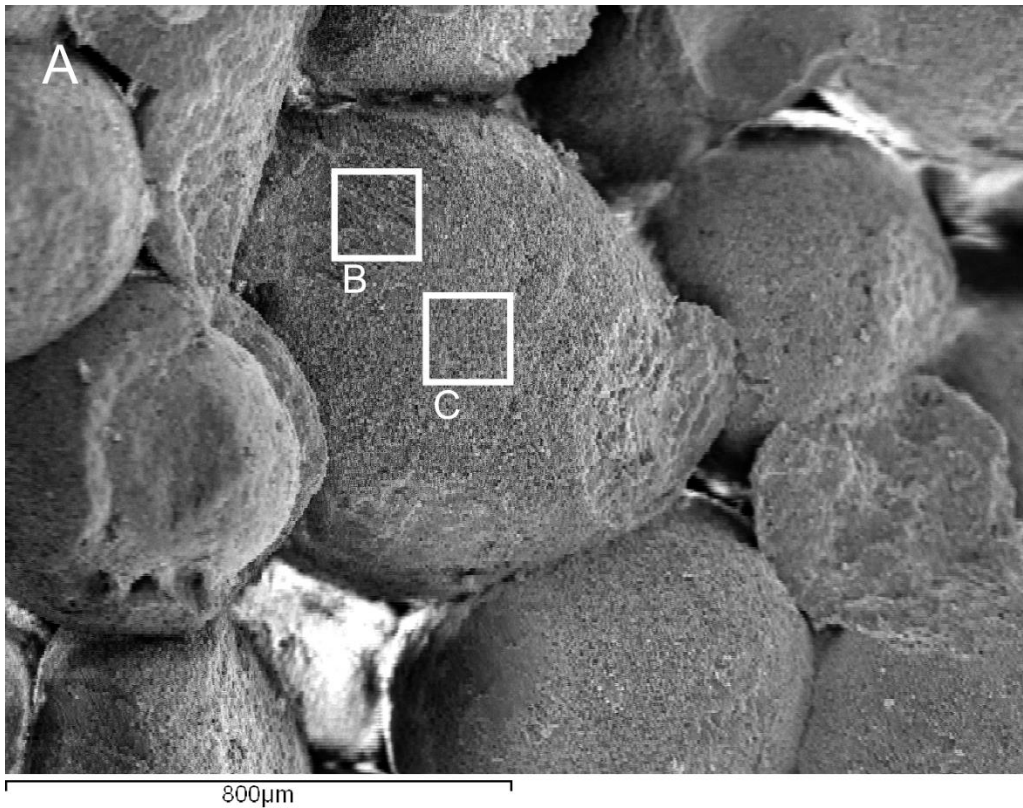
Receding



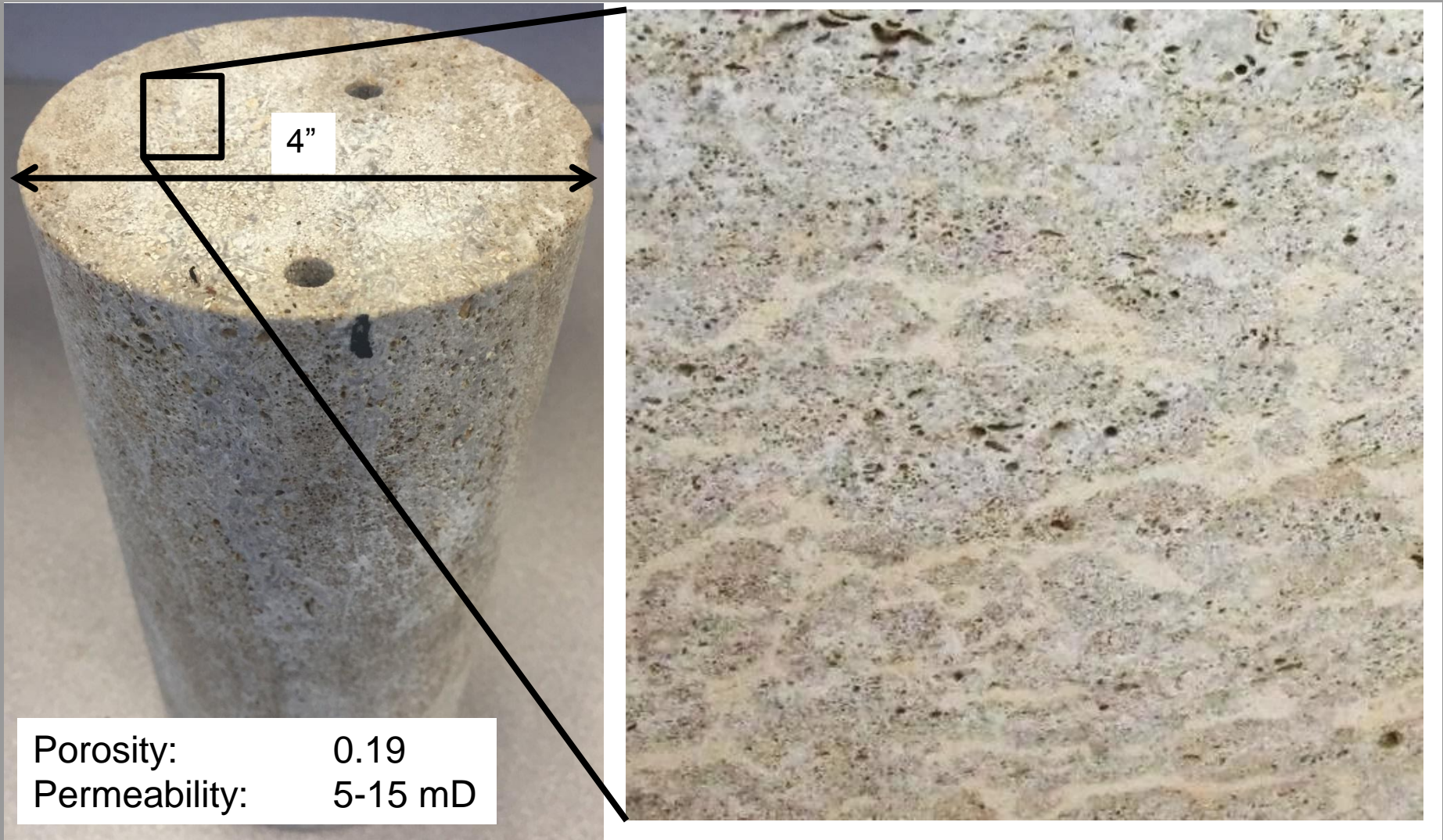
Contact angle distribution – contributing factors



Contact angle distribution – contributing factors



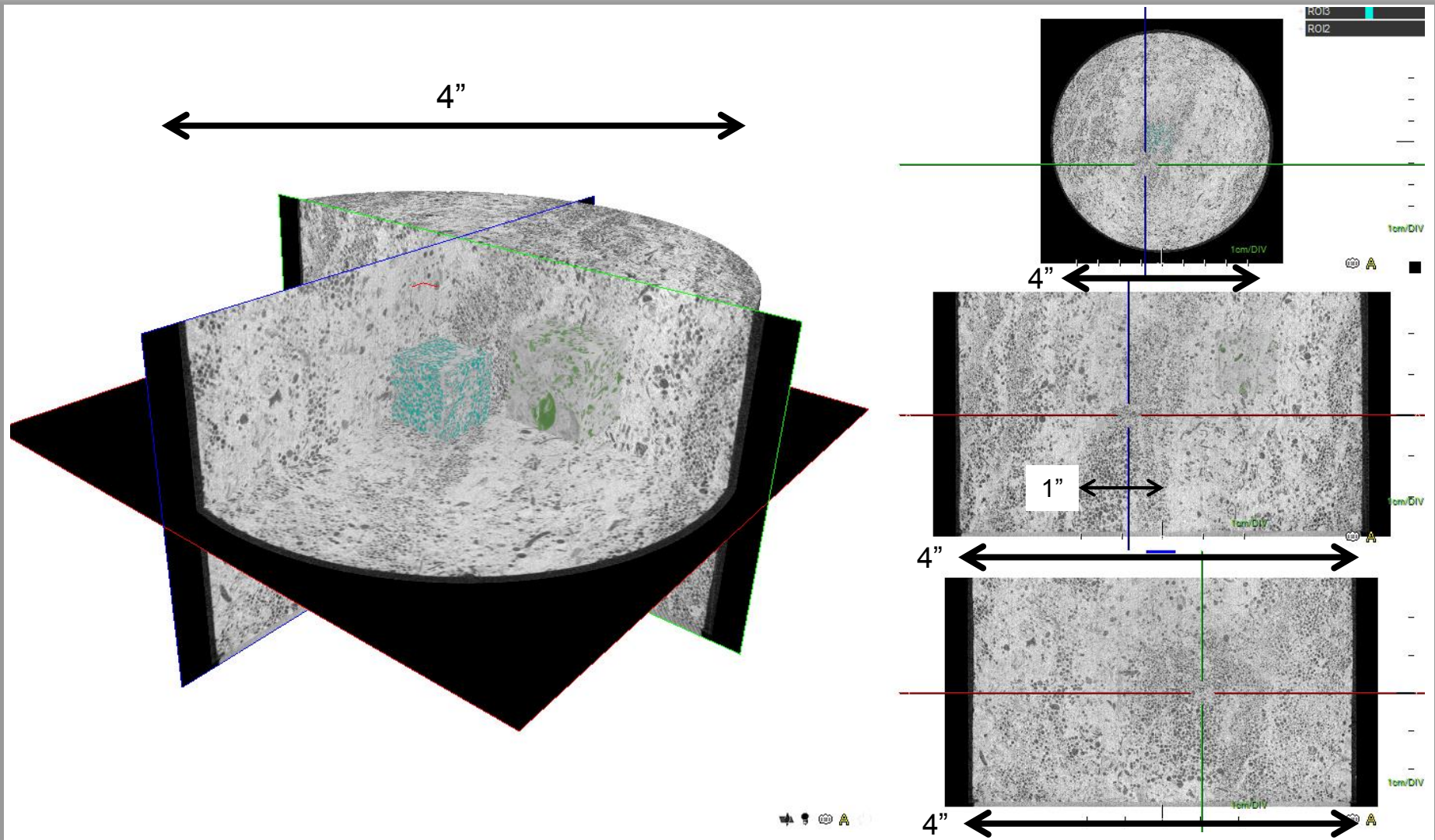
Heterogeneous Carbonate Sample



Porosity: 0.19
Permeability: 5-15 mD

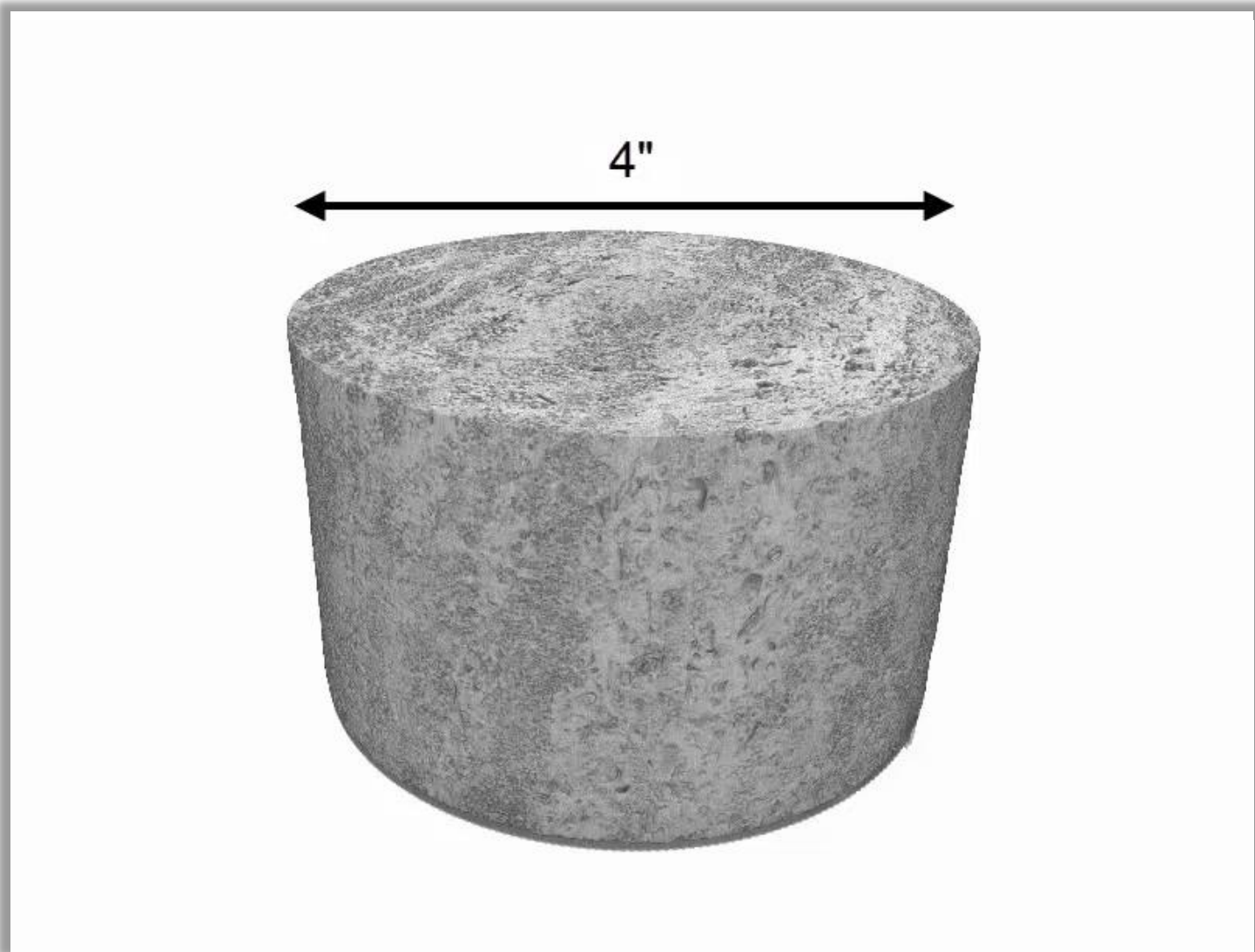
Results

Macroscopic Scanning



Results

Macroscopic Scan - Interpretation

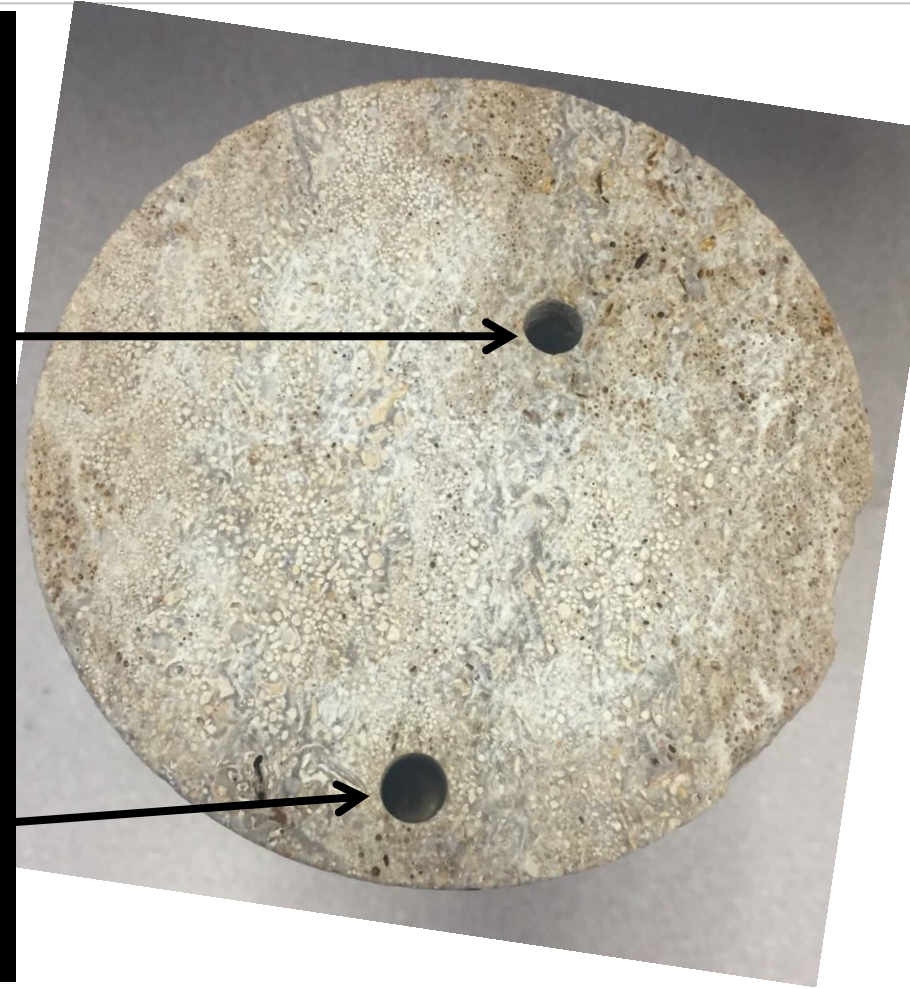
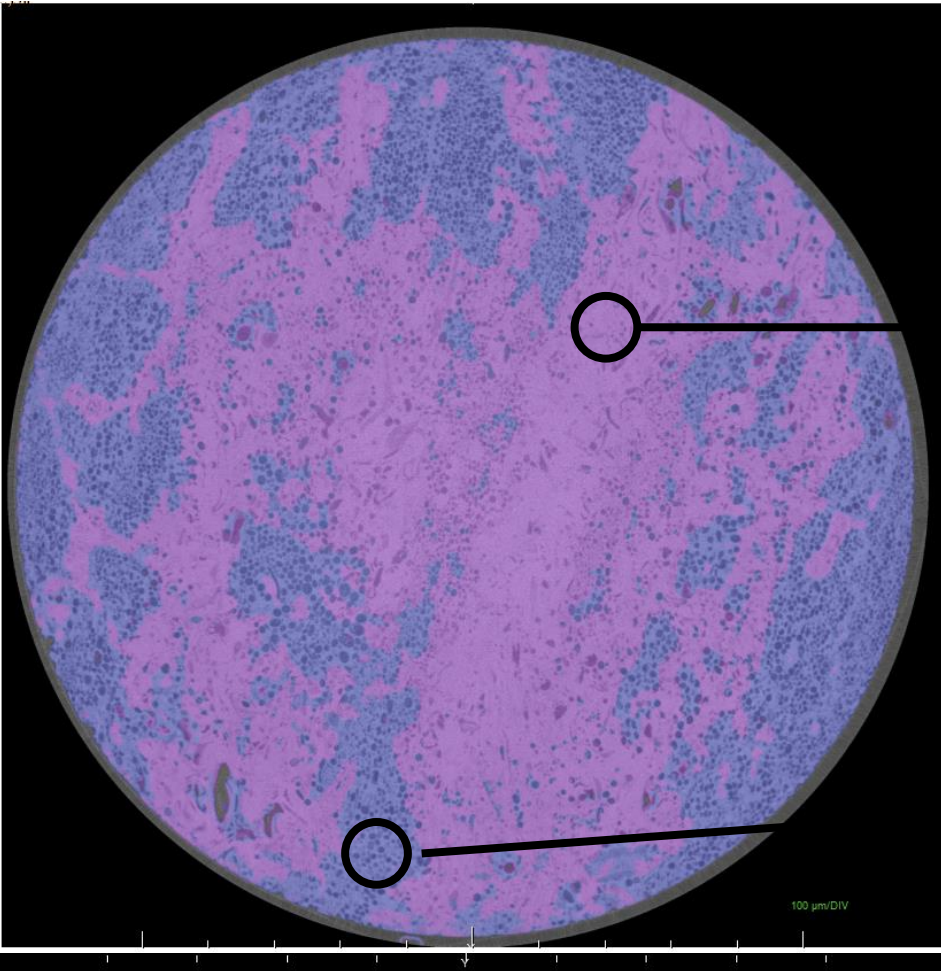


Results

Macroscopic Scan - Classification



Multi-scale Techniques – Mechanical Sampling

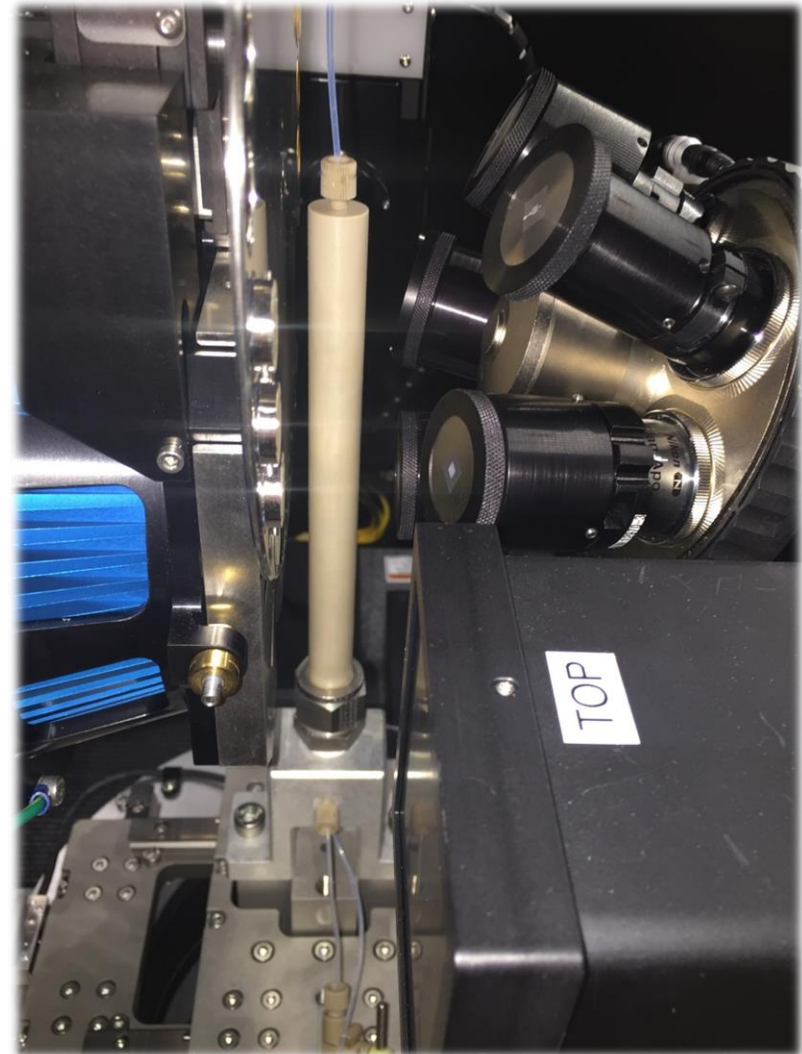
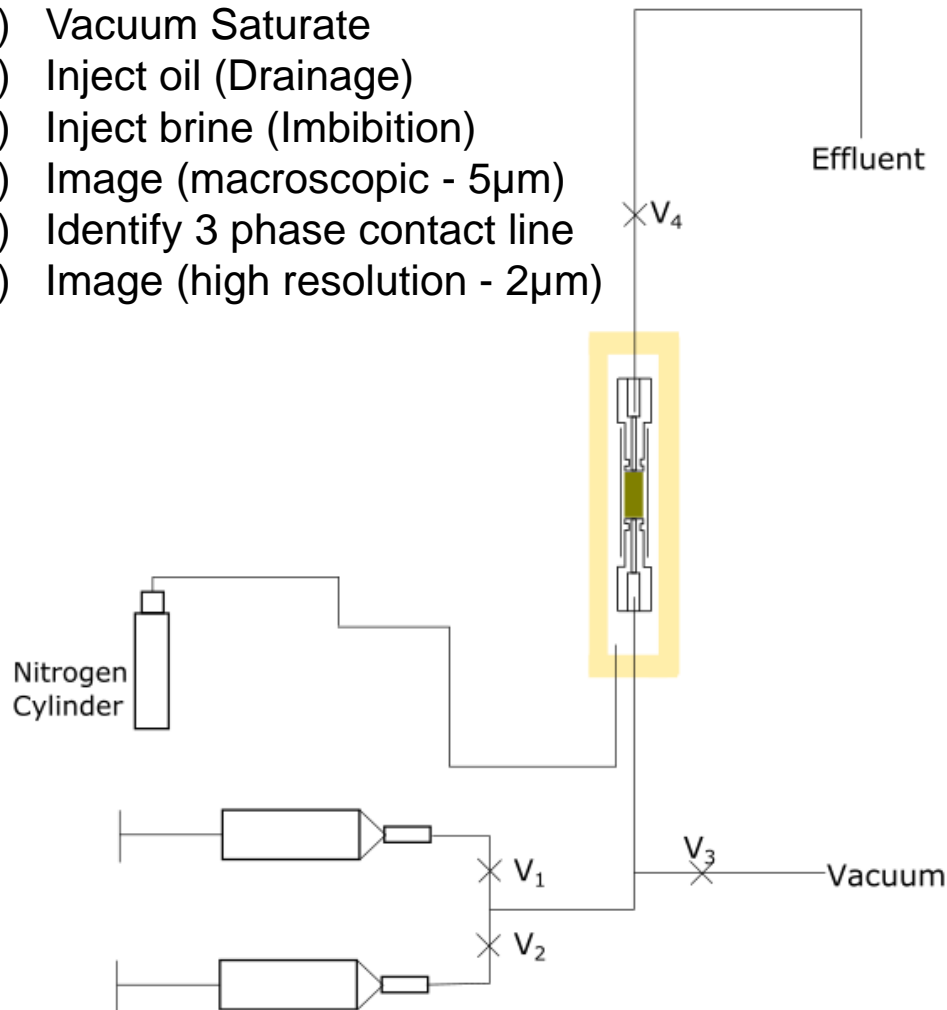


In Situ Experimentation

Custom Rigs & the Integration Kit



- 1) Vacuum Saturate
- 2) Inject oil (Drainage)
- 3) Inject brine (Imbibition)
- 4) Image (macroscopic - $5\mu\text{m}$)
- 5) Identify 3 phase contact line
- 6) Image (high resolution - $2\mu\text{m}$)

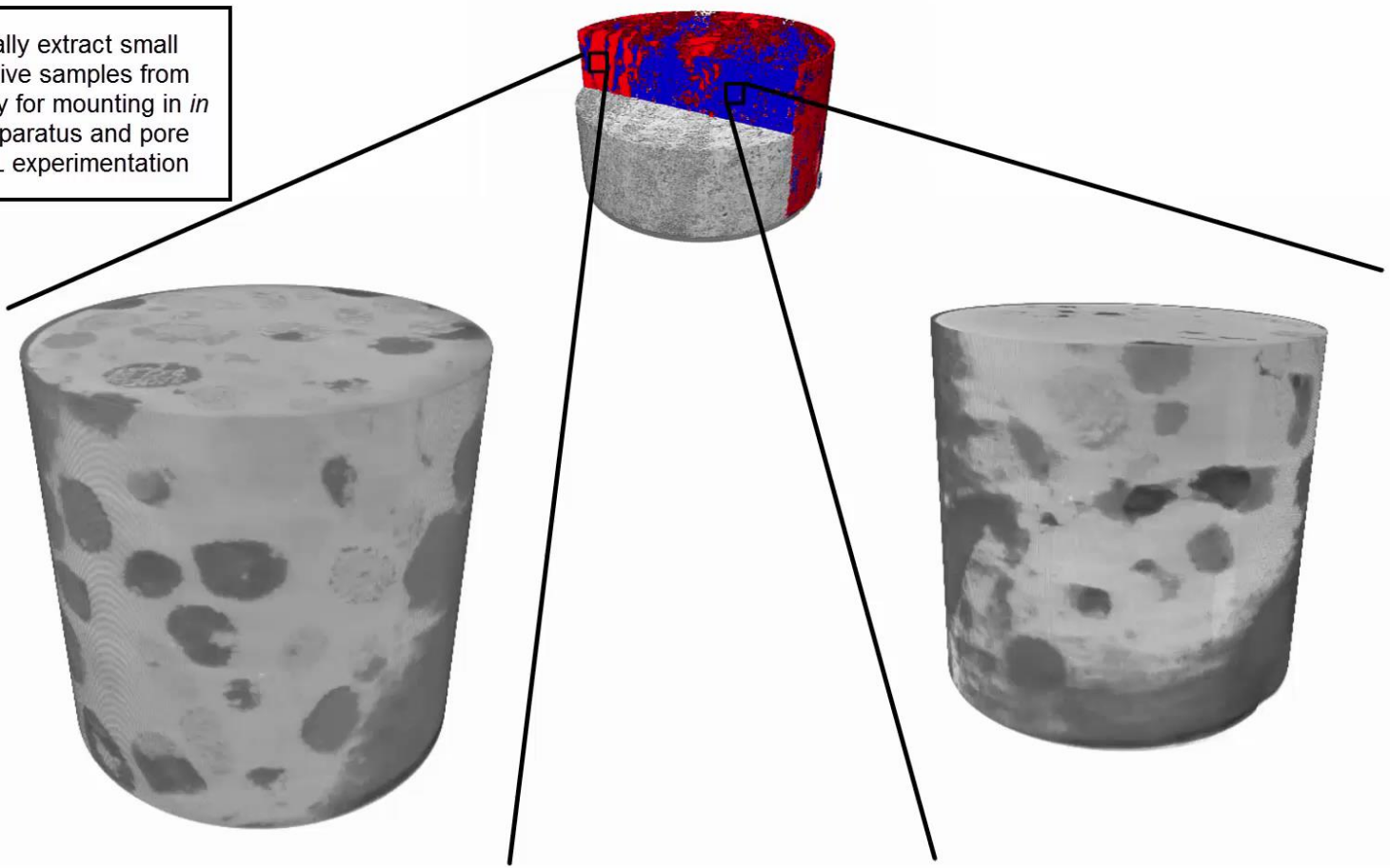


Results:

Pore-scale Wettability of Multiple Lithologies



Mechanically extract small representative samples from each lithology for mounting in *in situ* flow apparatus and pore scale SCAL experimentation



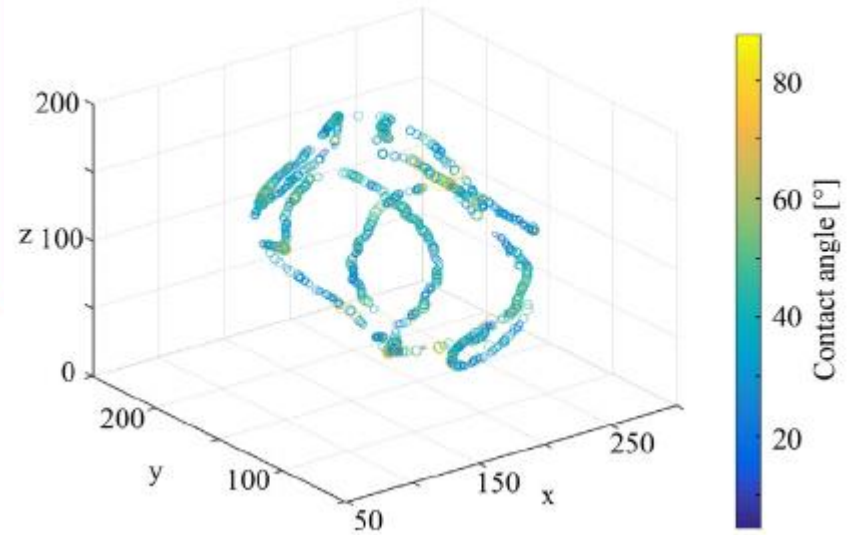
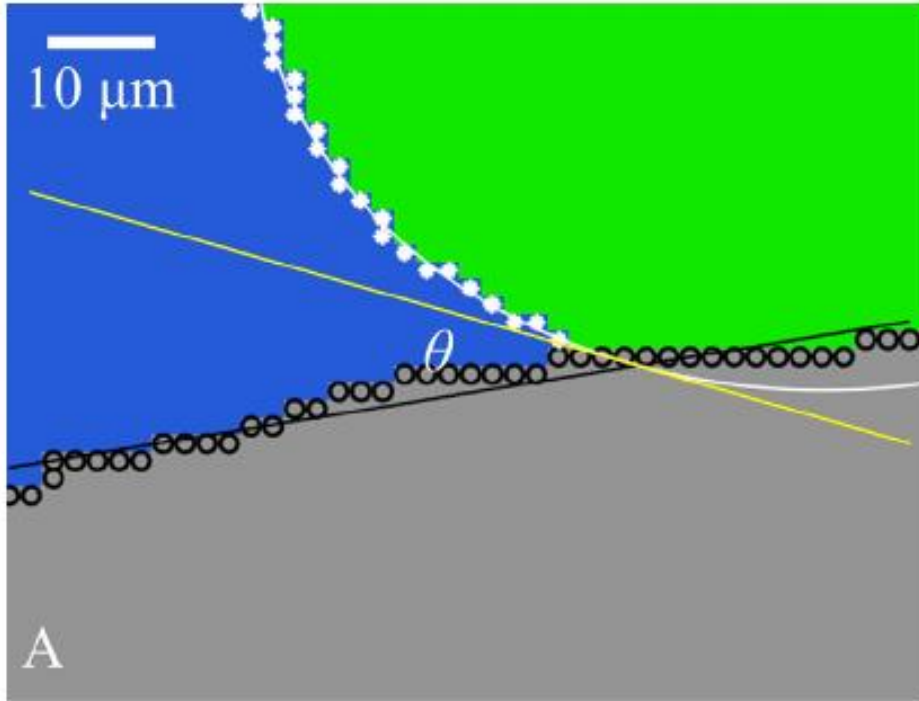
High porosity lithology

Low porosity lithology

Causes & Correlative Microscopy



Towards automation



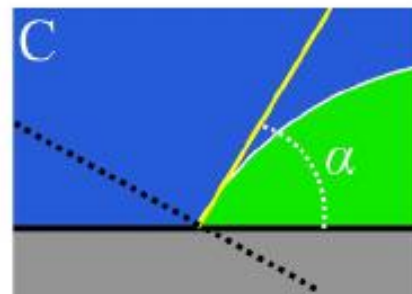
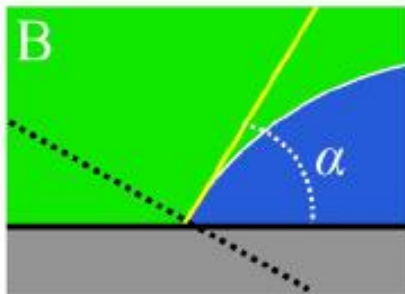
Automatic method for estimation of *in situ* effective contact angle from X-ray micro tomography images of two-phase flow in porous media

Alessio Scanziani^{a,b,*}, Kamaljit Singh^b, Martin J. Blunt^{a,b}, Alberto Guadagnini^{a,c}

^a Politecnico di Milano, Dipartimento di Ingegneria Civile e Ambientale, Piazza Leonardo da Vinci 32, 20133 Milan, Italy

^b Imperial College London, Department of Earth Science and Engineering, London SW7 2AZ, UK

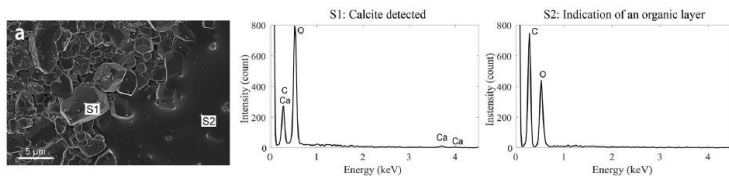
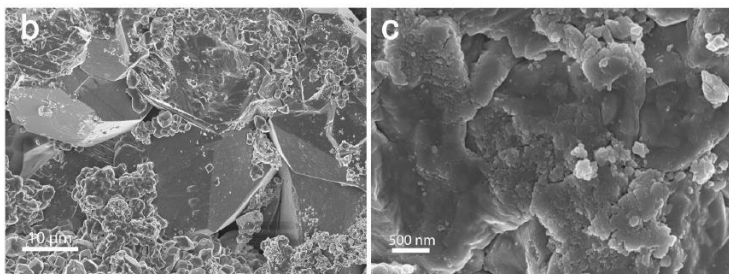
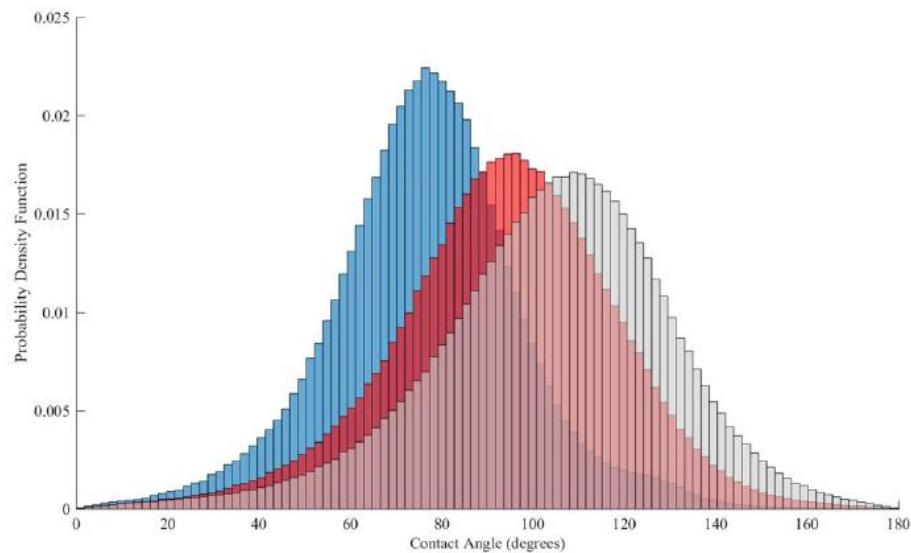
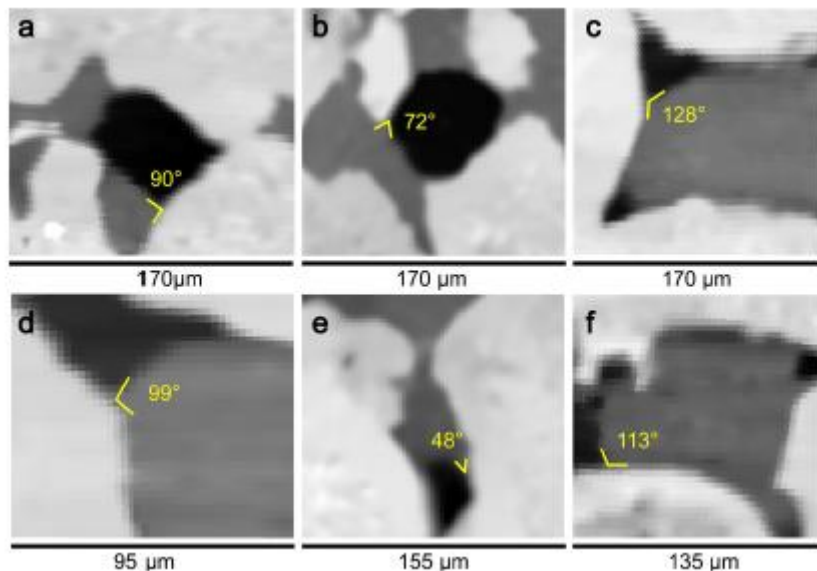
^c University of Arizona, Department of Hydrology and Atmospheric Science, Tucson, AZ 85721, USA



Automated contact angle estimation for three-dimensional X-ray microtomography data

Katherine Klise^a, Dylan Moriarty^a, Hongkyu Yoon^a, Zuleima Karpyn^b

Towards Automation Real Systems



SCIENTIFIC REPORTS

OPEN *In situ* characterization of mixed-wettability in a reservoir rock at subsurface conditions

Amer M. Alhammadi, Ahmed AlRatrou, Kamaljit Singh, Branko Bijeljic & Martin J. Blunt

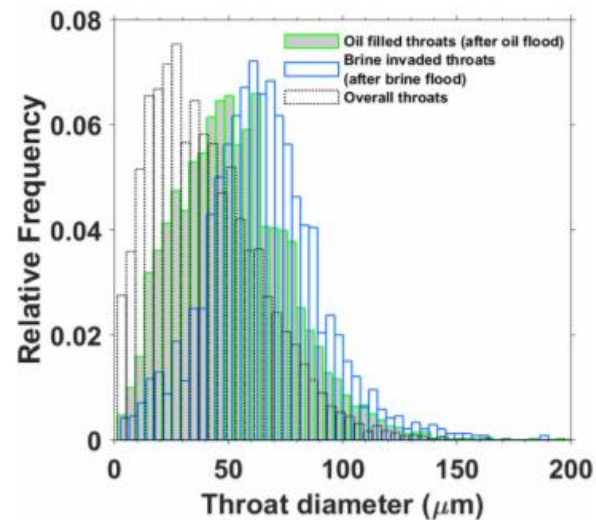
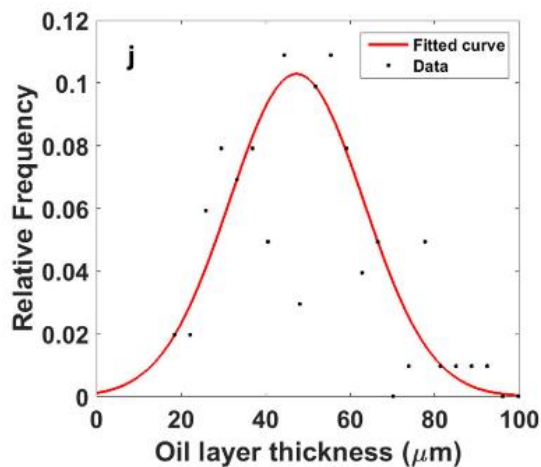
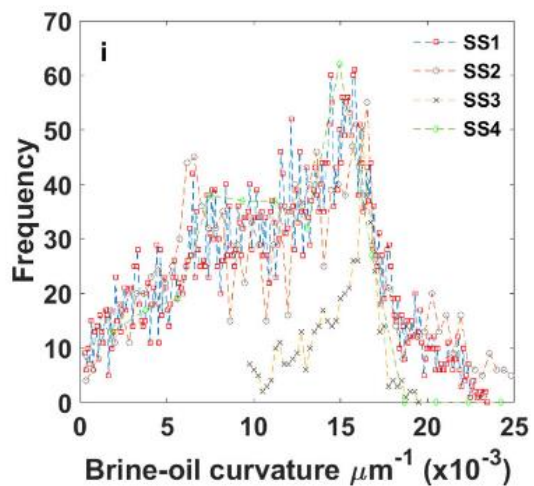
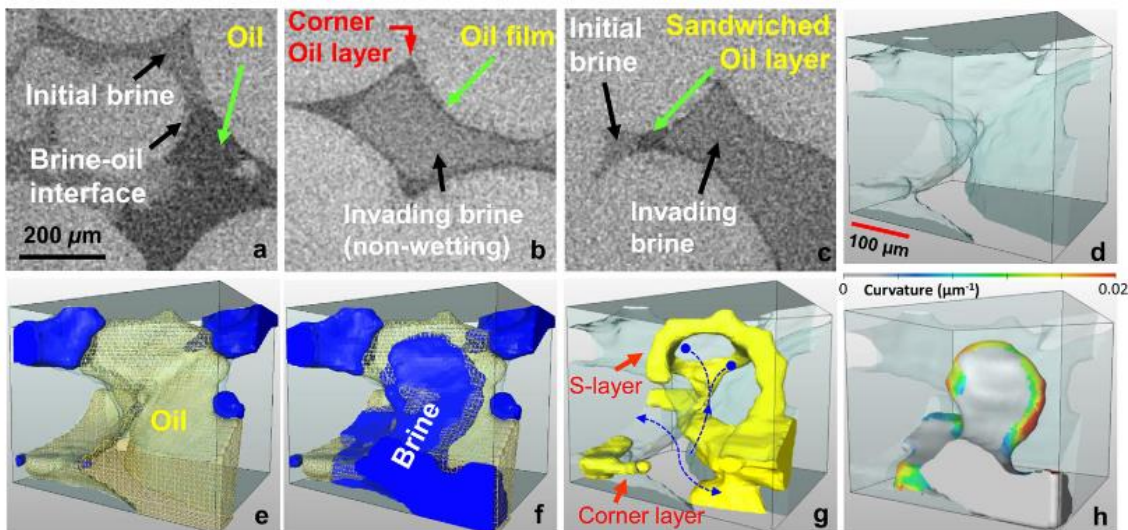
rd: 13 June 2017
ref: 17 August 2017

Applications

Oil layer measurement

Imaging of oil layers, curvature and contact angle in a mixed-wet and a water-wet carbonate rock

Kamaljit Singh¹, Branko Bijeljic¹, and Martin J. Blunt¹



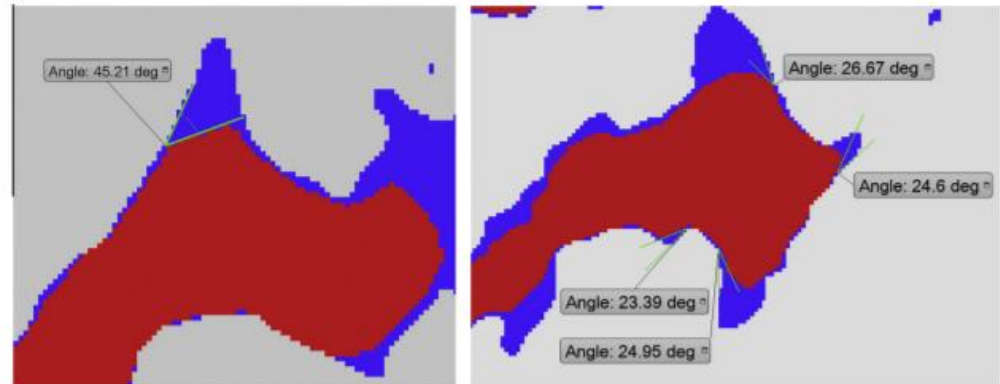
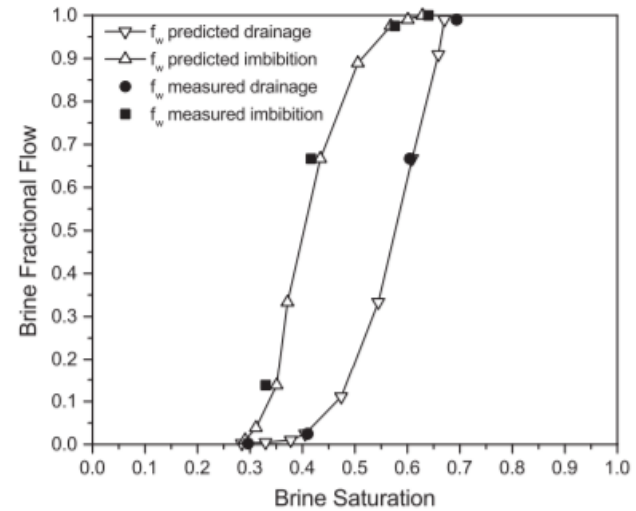
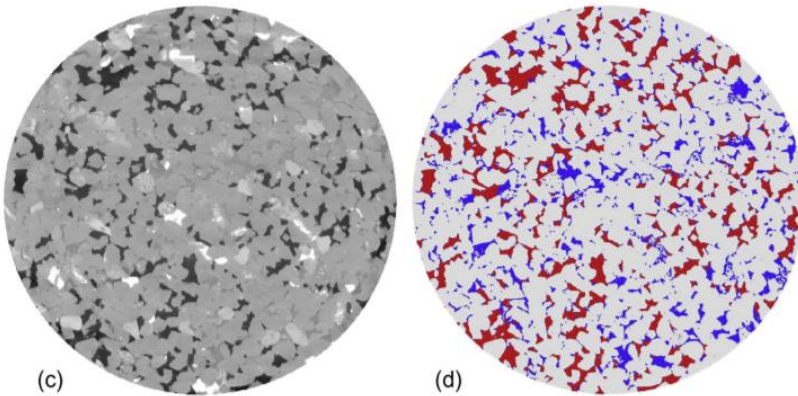
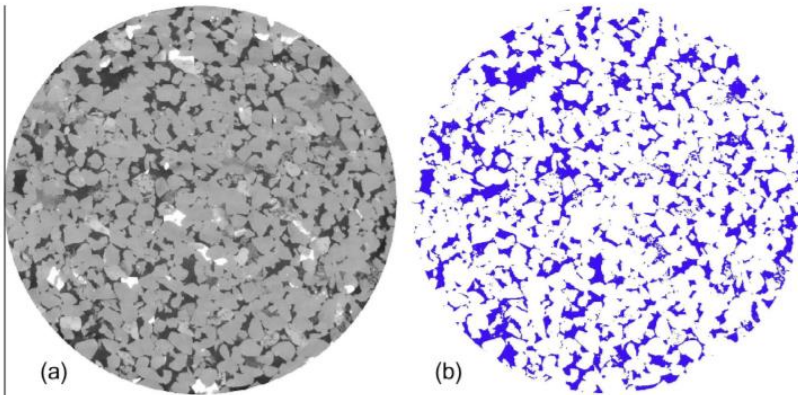
Applications

During steady state flow



Direct pore-to-core up-scaling of displacement processes:
Dynamic pore network modeling and experimentation

Arash Aghaei, Mohammad Piri *



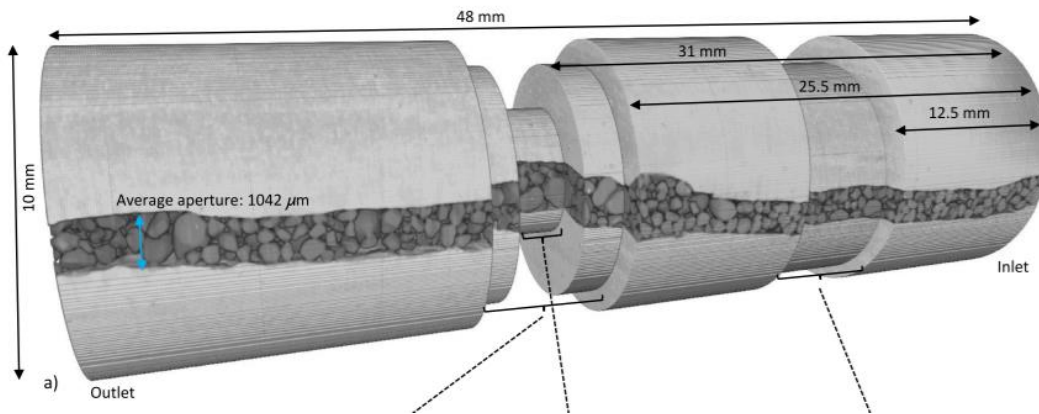
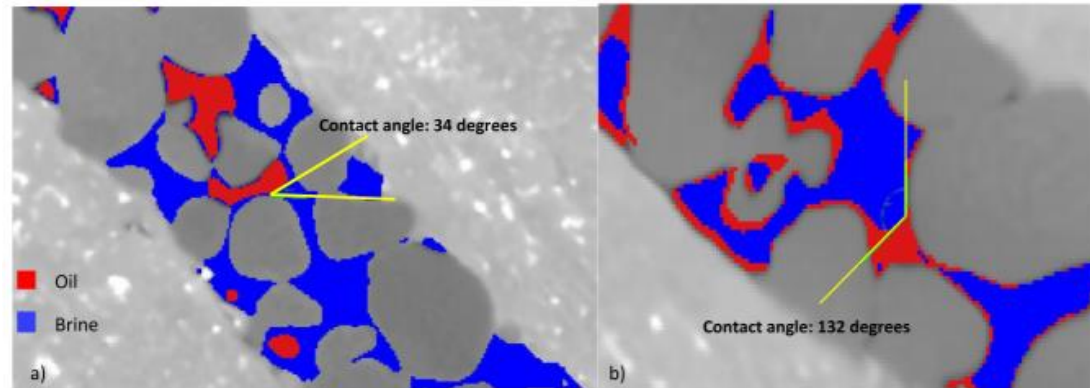
Applications

Proppant embedment



The effect of deformation on two-phase flow through proppant-packed fractured shale samples: A micro-scale experimental investigation

Maziar Arshadi^{a,*}, Arsalan Zolfaghari^a, Mohammad Piri^a, Ghaithan A. Al-Muntasheri^b, Mohammed Sayed^c



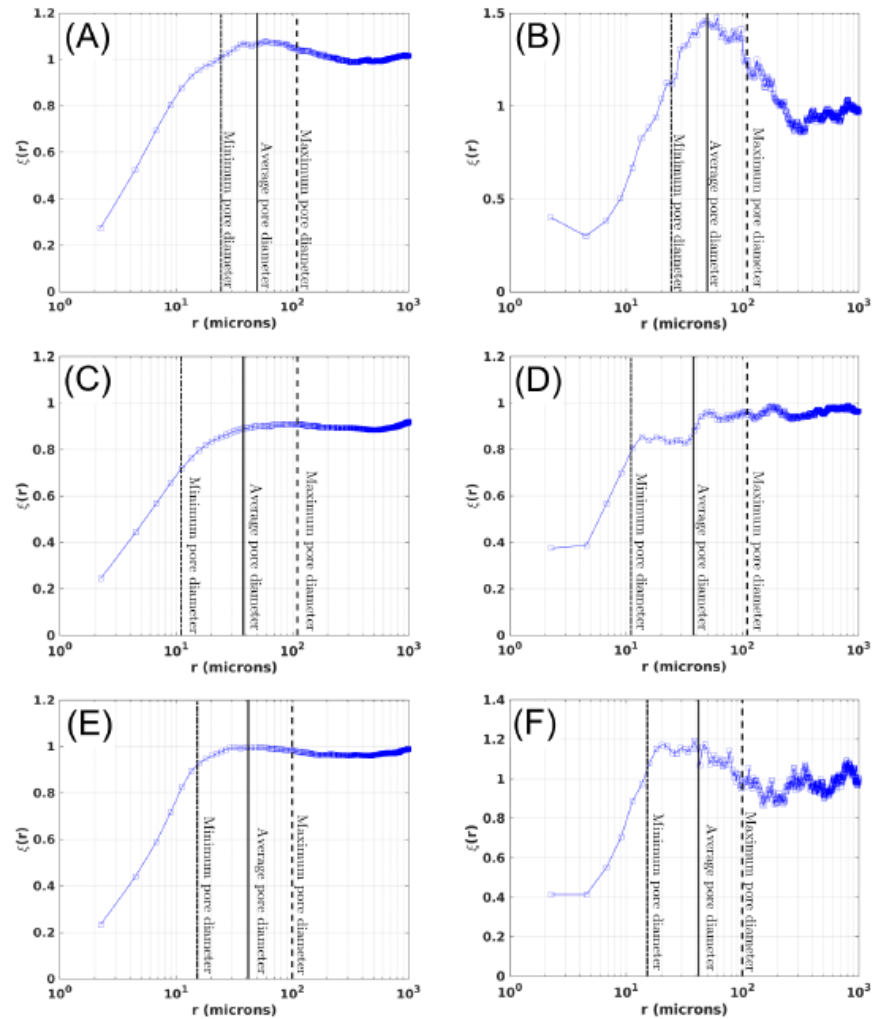
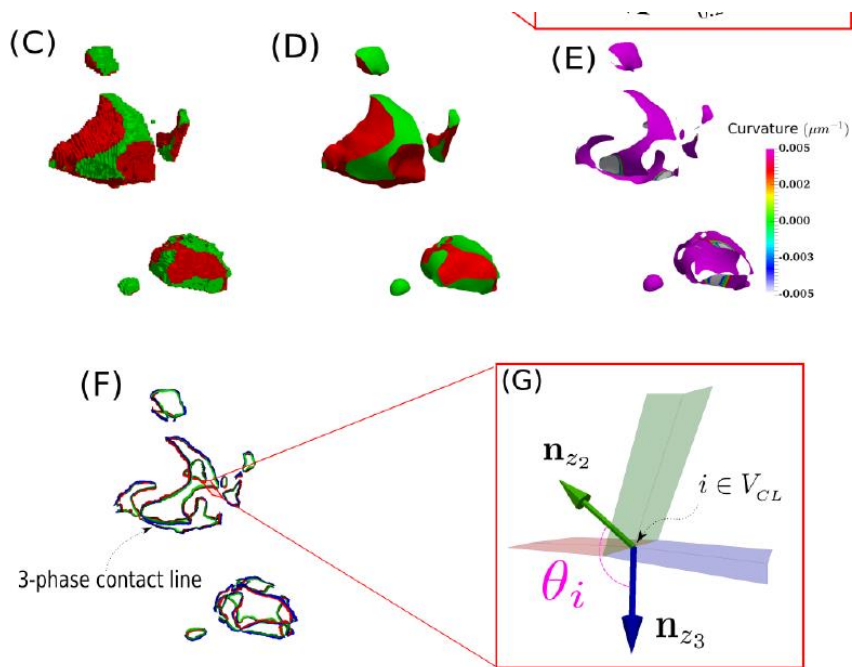
Applications

Contact angle and curvature

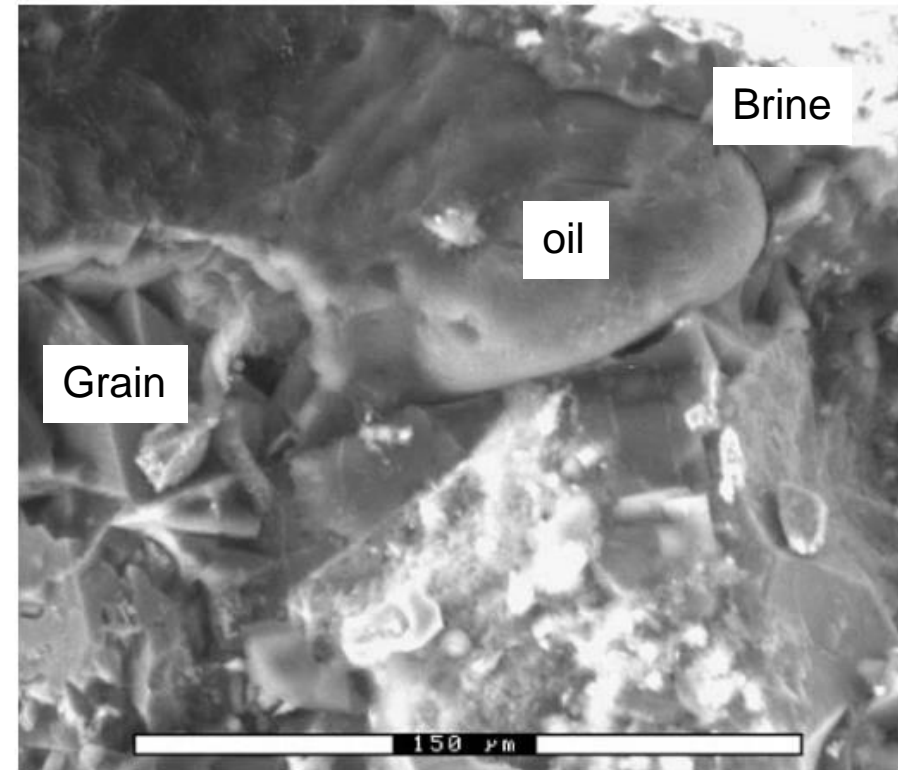
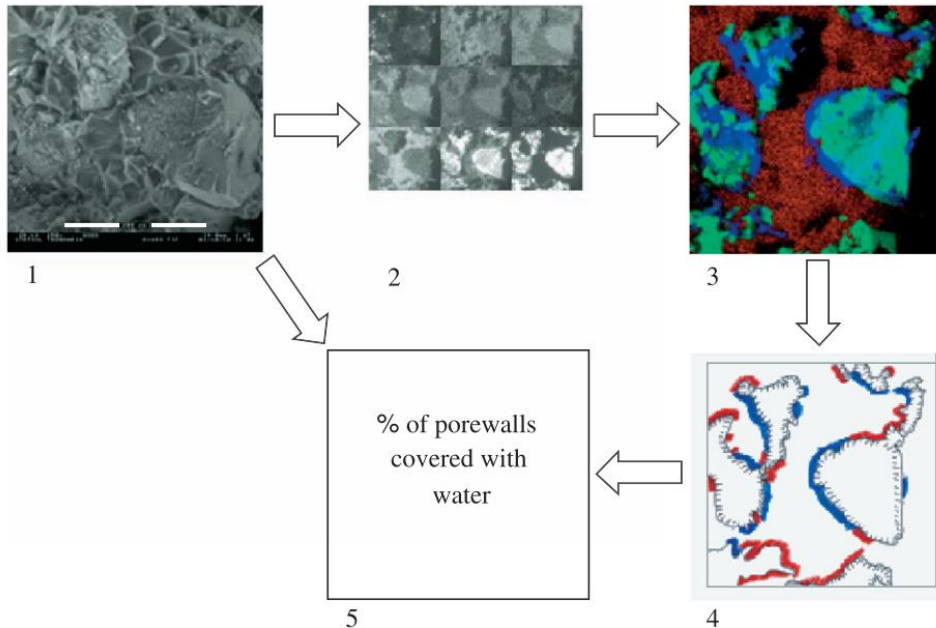
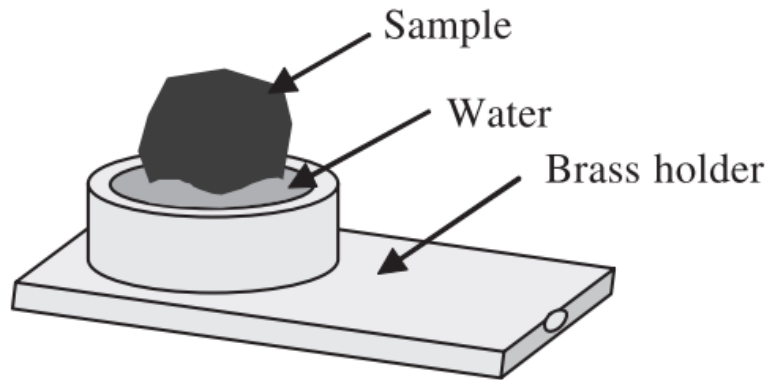


Spatial correlation of contact angle and curvature in pore-space images

Ahmed AlRatrou, Martin J. Blunt, and Branko Bijeljic

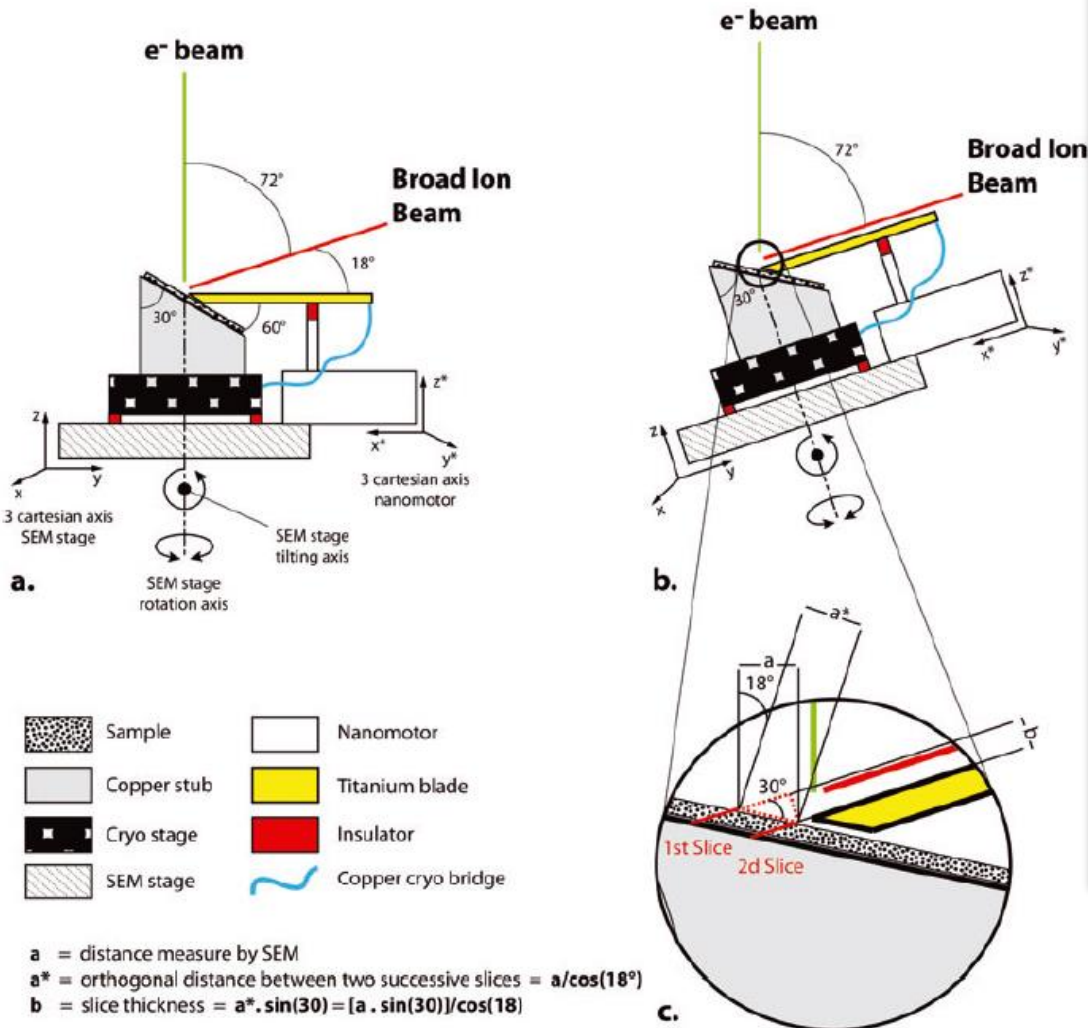


cryo-SEM - origins



Kowalewski et al. 2003, Journal of Petroleum Science and Engineering, Volume 39, Page 377, DOI: 10.1016/S0920-4105(03)00076-7

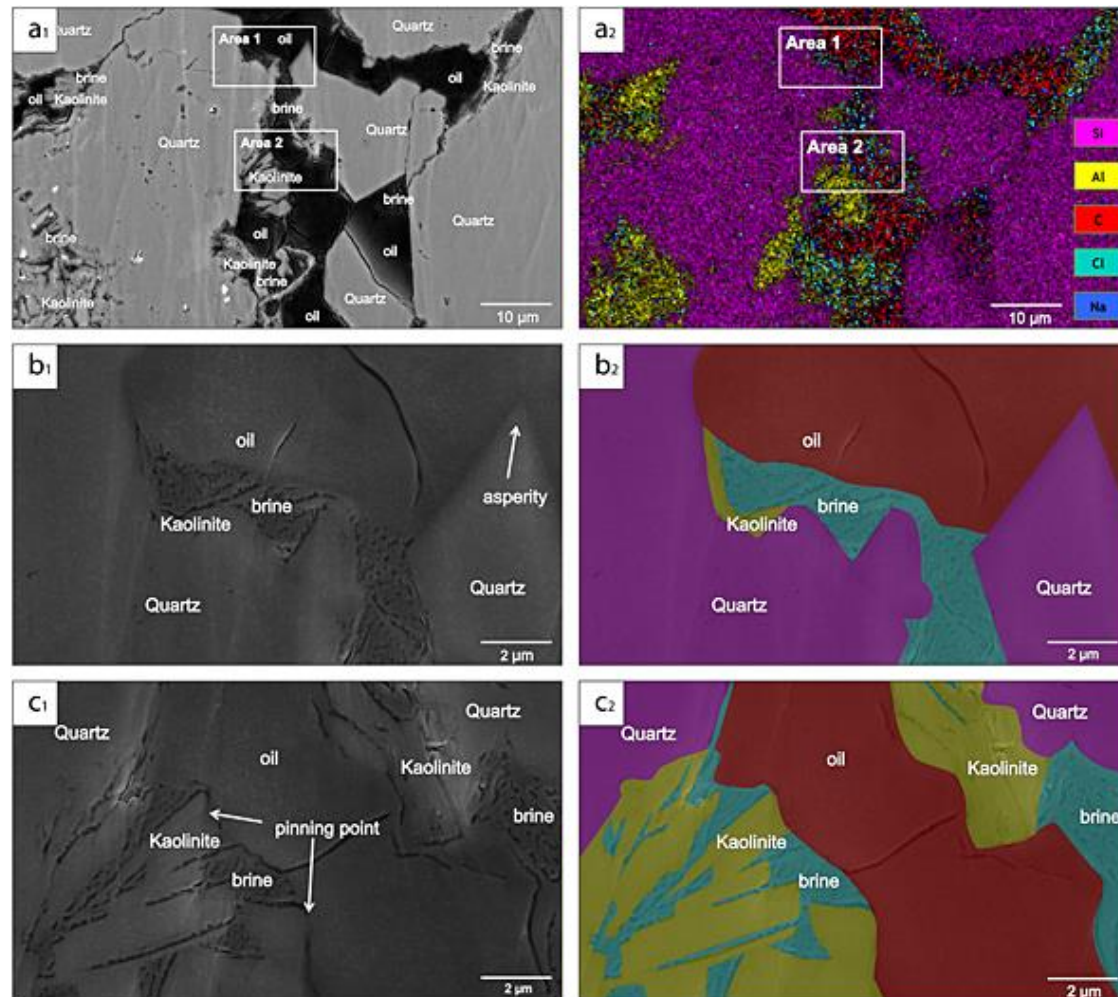
Imaging fluid occupancies using cryo-SEM



- ZEISS SUPRA SEM + custom Cryo BIB system
- Noble gas (argon) Broad Ion Beam used to mill sample
- Serial sectioning enabled by coupled sample stage, milling beam and imaging system
- Titanium BIB mask progressively withdrawn at sequential imaging steps to give high Z thickness accuracy
- Can be coupled with other EM techniques (e.g. EDX mapping) to give quantitative 3D chemical / mineral distributions

Desbois et al. 2013, *Journal of Microscopy*,
 Volume 249, Issue 3, pages 215-235
 DOI: 10.1111/jmi.12011

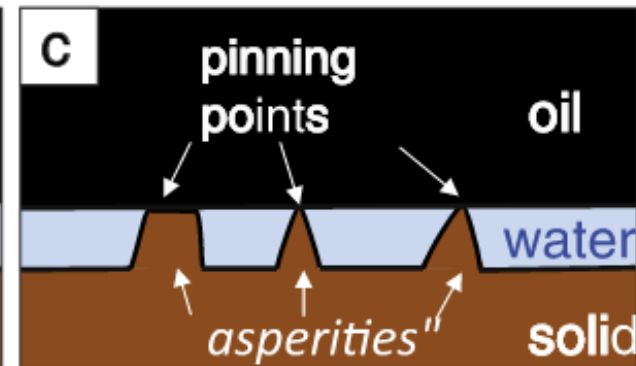
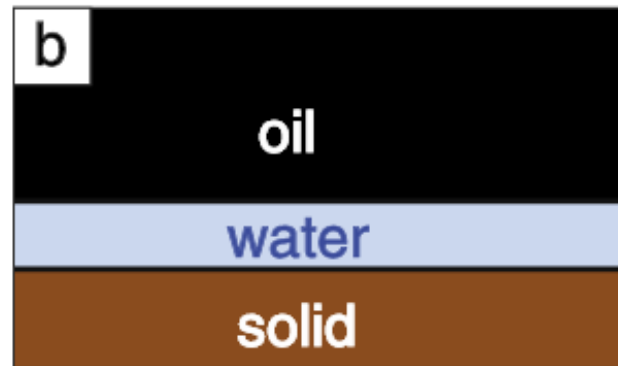
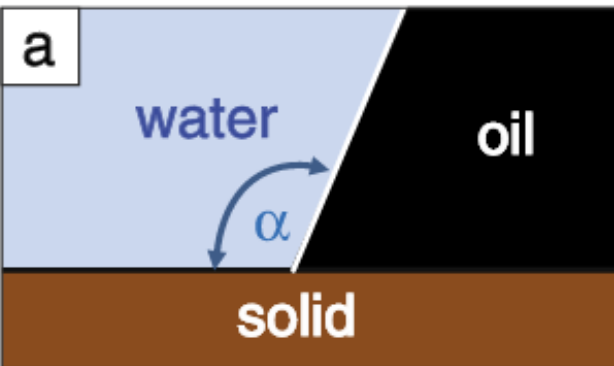
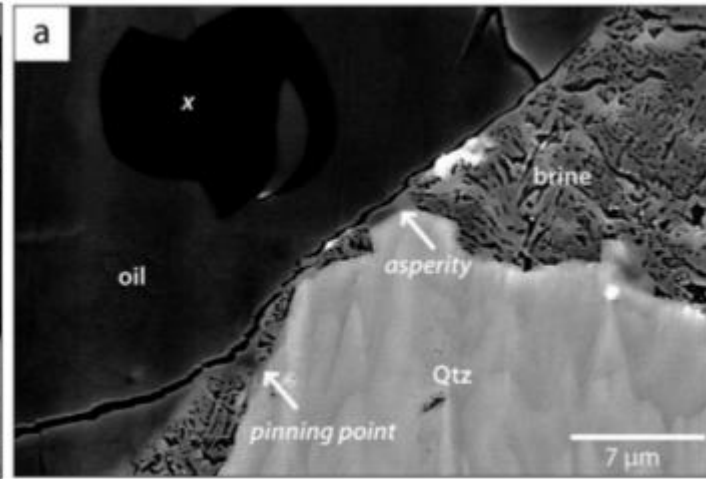
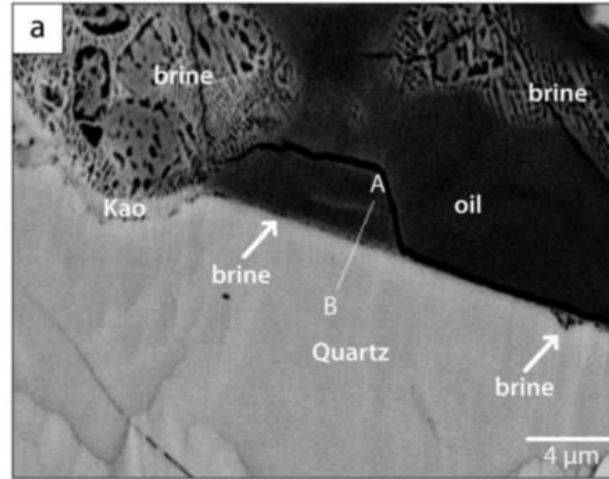
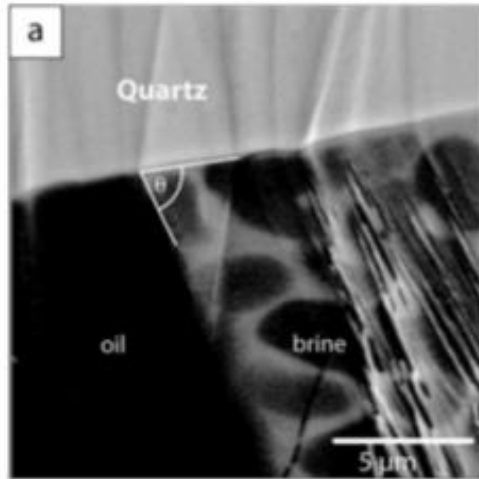
Imaging fluid occupancies using cryo-SEM



- Pixel Size: 60nm
- Fluids: NaCl Brine, Brent Crude,
- Sample: Obernkirchener Sandstone
- Quantitative EDX mapping used to map distribution of:
 - Oil
 - Brine
 - Clay
 - Quartz
- Three wetting conditions identified:
 1. Classical contact and contact angle
 2. Oil separated from rock by water film
 3. Local contacts via pinning through asperities – geometrical / chemical heterogeneity

Schmatz et al. 2015, *Geophysical Research Letters*,
 Volume 42, Issue 7, pages 2189-2195
 DOI: 10.1002/2015GL063354

Imaging fluid occupancies using cryo-SEM



Schatz et al. 2015, Geophysical Research Letters,

Volume 42, Issue 7, pages 2189-2195

DOI: 10.1002/2015GL063354

Matthew Andrew, PhD

